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**ANTHONY  
ASQUITH**

Commissioner of Patents  
PO Box 1450  
Alexandria, VA 22313-1450

16 April 2004

**Patent Attorney**

Dear Sir,

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**SUBMISSION OF PRIORITY DOCUMENTS**

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Serial Number: 10/662,818

Confirmation Number: 1104

Examiner: (not yet known)

Art Unit: 1724

Applicant: Gene Sidney SHELPS et al.

Our File Ref: 616-76US

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We now submit the certified copy of the priority document in respect of the above patent application.

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INVESTOR IN PEOPLE

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I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears an amendment, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

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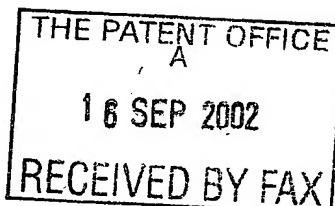
Signed

Dated 12 March 2004



# Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)



The Patent Office

Cardiff Road  
Newport  
Gwent NP9 1RH

1. Your reference

616-75GB

2. Patent application number

(The Patent Office will fill in this part)

0221439.3

16 SEP 2002

3. Full name, address and postcode of the or of each applicant (underline all surnames)

ENPAR Technologies  
incorporated.  
449 Laird Road, unit 12  
Guelph, Ontario  
N1G 4W1

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

CANADA

PA 16/9/02

077 85801001

4. Title of the invention

Ion-Exchange / electrochemical  
Treatment of ammonia in waste-water

5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Anthony Asquith + Co  
328 Leeds Road  
Scholes  
Leeds  
LS15 4DD

Patents ADP number (if you know it)

06562201003

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number  
(if you know it)

Date of filing  
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing  
(day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

- a) any applicant named in part 3 is not an inventor, or
- b) there is an inventor who is not named as an applicant, or
- c) any named applicant is a corporate body.

See note (d))

# Patents Form 1/77

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Continuation sheets of this form

Description 6

Claim(s) 2

Abstract 1

Drawing(s) 2 only

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11. I/We request the grant of a patent on the basis of this application.

Signature

Date

12. Name and daytime telephone number of person to contact in the United Kingdom

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Title: ION-EXCHANGE / ELECTROCHEMICAL TREATMENT OF AMMONIA IN WASTE-WATER

[001] This invention relates to the breakdown of dissolved ammonia and ammonium, in waste-water, and to the desideratum of discharging the resulting nitrogen in the form of nitrogen gas. It is known that treatment of ammonium-water by electrolysis can release the nitrogen in the form of nitrogen gas, and the present invention aims to improve the cost-effectiveness of that electrolysis technology.

BACKGROUND TO THE INVENTION

[002] The traditional non-electrolytic methods of breaking down and removing ammonia and ammonium from water have basically involved engineering a system which exposes the water to oxygen. The system promotes oxidation reactions, which usually have had a high aerobic micro-biological component. However, such oxidation reactions predominantly leave the nitrogen in the form of (dissolved) nitrate and of the various (gaseous) oxides of nitrogen. These nitrogen compounds themselves can be health hazards, and should not be simply discharged into the environment, at least not in the large quantities that arise from treating waste-water.

[003] Patent publication US-6,083,377 (Lin + Shelp, July 2000) describes the use of electrolysis, in effecting the direct breakdown of ammonium to nitrogen gas. Effective though it can be, under many conditions, the disclosed electrolysis technology works best when the concentration of ammonium is high. When the concentration is low, the amount of electrical energy needed to drive current through the electrolytic cells can spoil the economics of the process. If the energy usage is kept economical, the ammonium content of the discharged water, though diminished, still might be too high.

[004] It is an aim of the present invention to provide (a) that the ammonium in waste-water is converted, by electrolysis, directly to nitrogen gas; (b) that the concentration level of ammonia and ammonium in the discharged water is acceptably low; and yet (c) that only an economic amount of electrical energy is required.

[005] Breaking down ammonium by electrolysis, when properly engineered and operated, avoids the formation of the unwanted and toxic oxidation products. Also, it is recognised that electrolytic treatment is much less dependent on ambient temperature than aerobic micro-biological oxidation. That is to say, electrolysis would be preferred for cold climates, for achieving the required diminishment of ammonium, if it could be done more economically.

THE INVENTION IN RELATION TO THE PRIOR ART

[006] It is well known that dissolved ammonium can be removed from waste-water by ion-exchange. A solid body of sorbent material is provided, and the waste-water containing the ammonium passes through the body. The ammonium is sorbed out of solution in exchange for a cation (such as sodium), and the body retains the ammonium ions on the material.

[007] When the ion-exchange medium becomes saturated with ammonium, now the ammonium has to be removed therefrom. The flow of waste-water is shut off, or diverted away from the sorbent body, and a volume of regenerant water is flushed through the sorbent body. The regenerant water contains a high concentration of dissolved sodium chloride, and the sodium ions displace the ammonium ions sorbed on the sorbent body. The ammonium ions then pass into solution in the regenerant water.

[008] After flushing, the waste-water flow can be fed through the sorbent body, which will once again act to sorb ammonium out of the waste-water. After flushing, the volume of regenerant water, with the ammonium now dissolved therein, drains back into a regenerant storage tank. The ammonium remains, still untreated, in the regenerant water, although at a higher concentration level than the concentration in the waste-water.

[009] It is recognised that the ammonium in the regenerant water, being more concentrated than the ammonium in the waste-water, is easier to treat. The higher the concentration, the greater the rate at which the break-down reactions can take place, and this is true even when the ammonium is to be removed by the traditional aerobic microbial oxidation methods. However, it is recognised, in the invention, that the higher concentration of ammonium in the regenerant water makes it hugely more economical to extract the ammonium from the regenerant water by electrolysis, than it was to extract the ammonium from the waste-water itself, directly, by electrolysis.

[0010] It is noted that the regenerant water contains sodium chloride, or some similar salt, in high concentration. It is recognised that the presence of such salt in the regenerant water means that the regenerant water is much

more electrically-conductive than the waste-water. Thus, the energy needed to electrolyse the regenerant water, simply because the regenerant water is salty, is much diminished, compared with the energy needed to electrolyse the waste-water itself, directly.

[0011] The regenerant water is kept in a storage tank, between flushings. During regeneration, the regenerant water is circulated through an electrolytic cell, whereby the ammonium in the regenerant water is broken down. Preferably, the cell is operated in such manner that the redox voltage of the electrolyte, i.e. of the regenerant water, and the pH of the regenerant water, are such that the ammonium breaks down in such manner that the nitrogen component of the ammonium takes the form of nitrogen gas. The said US-8,083,377, to which attention is hereby directed, shows how to operate an electrolytic cell such that the desired conditions of Eh and pH can be realised, which will lead to the formation of nitrogen gas.

[0012] It is recognised that the amount of electrical energy needed to break ammonium down to nitrogen gas is well within the limits of what is economically practical, under a wide range of conditions, when the electrolysis is done on the regenerant water. By contrast, when the electrolysis was done on the waste-water directly, only under a narrow range of conditions could the system be run economically. The invention aims to provide a system in which the treated waste-water is discharged with only a negligibly-low ammonium content, in which the extracted ammonium is converted directly into nitrogen gas, and in which the system can be run economically under a wide range of conditions.

[0013] As will be explained, the regenerant water, though subject to occasional make-up of some of its constituents, basically is unchanged by the ion-exchange / electrolysis cycle, and can be recycled indefinitely. This may be contrasted with many traditional ion-exchange systems, in which the regenerant water is used once, to capture the contaminant, and then discarded with the contaminant still intact.

[0014] In the present case, the combination of the ion-exchange, with electrolysis, to treat ammonium, means that: (a) the contained volume of regenerant water can be tailored to the requirements of efficient electrolysis much more readily than can a stream of waste-water; (b) the fact that the regenerant water is of high ionic strength means that the electrical resistance of the water is low, which leads to efficient electrolysis; and (c) the overall ammonium reaction leaves the regenerant water chemically unchanged, i.e. as it was before the ammonium was captured, and thus able to be recycled.

#### DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

[0015] By way of further explanation of the invention, exemplary embodiments of the invention will now be described with reference to the accompanying drawings, in which:  
Fig 1 is a diagram showing elements of apparatus that have been combined and configured to operate in the manner of the invention.

Fig 2 is a diagram of a municipal waste-water treatment plant, which includes the apparatus of Fig 1.  
Fig 3 is a diagram showing some aspects of the control of a system that embodies the invention.

[0016] The apparatuses shown in the accompanying drawings and described below are examples which embody the invention. It should be noted that the scope of the invention is defined by the accompanying claims, and not necessarily by specific features of exemplary embodiments.

[0017] Fig 1 shows waste-water to be treated entering the apparatus 20 through an inlet port 4, and passing through an ion-exchange station 23, or sorption station, which includes ion-exchange columns 1,2,3. The waste-water may be passed through just one of the columns, or through more than one of the columns, in series. Ammonium in the waste-water is removed by ion-exchange, onto bodies of sorbent material in the columns. The treated waste-water then passes out of the treatment apparatus through a discharge port 5, substantially ammonia-, ammonium-free. A properly-engineered, yet economical, sorption station can be very effective at driving the ammonium level down to insignificant concentrations, in water discharged from the sorption station.

[0018] A notional sorption station may be compared with a notional electrolytic station, from the standpoint of driving the concentration level of ammonium, in the water discharged from the station, down to an acceptably low (i.e. safe) level. As mentioned, it is recognised, in that comparison, that the electrolytic station that would be needed, in order to get down to a given (low) concentration, is considerably less economical than the corresponding sorption station. On the other hand, of course, in the sorption station, the ammonium would remain intact, whereas in the electrolytic station the ammonium is converted directly to nitrogen gas.

[0019] The ion-exchange sorbent material may comprise naturally occurring materials such as zeolites, or

equivalent synthetic resins that function as cation exchangers. To the extent possible, the ion-exchange media should be so configured as to specifically target ammonium as the substance to be sorbed.

[0020] When the sorbent material in e.g. column 3 has become saturated with ammonium, water-flow control valves are operated, to divert the incoming waste-water stream away from the saturated column 3 to one of the other columns. The control valves are operated also to cause water from a regenerant tank 8 to circulate through the saturated column 3. The regenerant circulation through column 3 is indicated by the dotted arrows in Fig 1. The valves can be operated in such manner as to pass the regenerant water from the tank 8 through the other columns 1,2, of course, as and when those become saturated with ammonium.

[0021] The regenerant water from the tank 8, which flushes through the ion-exchange resin in the column 3, is water that has been charged with a high concentration of sodium chloride (i.e. the regenerant water is concentrated brine). The sodium ions displace the ammonium, on the sorbent material, in the column. The ammonium passes (back) into solution, but now into the regenerant water. The sodium ions remain on the sorbent. The flow of the regenerant water is controlled by the various valves and pumps, in the regenerant tank 8, in the electrolysis station 24, and elsewhere, as shown. While sodium chloride is satisfactory, and inexpensive, other salts may be used in the regenerant water, to provide the ion-exchange function, such as potassium and calcium chloride salts.

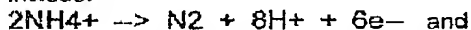
[0022] When the ion-exchange media in the column 3 has been flushed clear of ammonium, the control valves are again operated, to make the column once again open to the incoming flow of waste-water, and available for sorbing ammonium out of the waste-water. The now-flushed ion-exchange material can function, once again, to capture ammonium ions out of solution, i.e. out of the waste-water being treated.

[0023] The engineer may prefer to run the incoming waste-water through more than one of the columns, if more than one is available, running the waste-water first through the column that is most nearly saturated, and then through the less-saturated column.

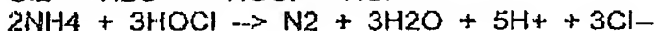
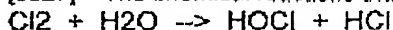
[0024] The regenerant water, upon returning from the ion-exchange column, contains a high concentration of ammonium. In accordance with the invention, the ammonium-laden regenerant water is circulated through the electrolysis station 24. Here, the ammonium is broken down, and the nitrogen component thereof is released as nitrogen gas, at 10. The ammonium in the regenerant water, i.e. the ammonium that has been flushed out of the sorbent in the column, is at a higher concentration in the regenerant water than it was in the waste-water passing through the columns, and again it is noted that electrical energy can be used to promote the ammonium breakdown reactions, by electrolysis, with much improved efficiency, if the ammonium concentration is high.

[0025] The breakdown reactions that take place in the electrolysis station 24 will now be considered. In the electrolysis station, the ammonium dissolved in the regenerant water is broken down at the anode into nitrogen gas. The reaction creates hydrogen ions and electrons. Chloride ions in the water also react at the anode, producing chlorine and electrons. The chlorine dissolves in and combines with water, to form hydrochloric and hypochlorous acids. Ammonium still present in the water then reacts especially with the hypochlorous acid, to create nitrogen (gas), and to release hydrogen ions and chloride ions into the water. (It is recognised that parallel reactions can be engineered using bromide and other halide salts.)

[0026] Thus, the main electrolytic reactions of interest are oxidation reactions that occur at the anode, which include:



[0027] The chemical reactions that result include:



[0028] These reactions are simple enough to engineer. It is possible to monitor and control the pH and the redox voltage of the regenerant water passing through the electrolytic cell, but it is recognised that the conditions can readily be engineered without close control. Once the conditions have been thermodynamically created in which the reactions take place, the tendency of the ammonium to transform directly to nitrogen gas is very strong. Very little of the unwanted oxidation products, such as nitrate and the various nitrogen oxides, are produced.

[0029] Thus, the ammonium in the regenerant water is disposed of, leaving nothing but nitrogen gas, and a little acidity in the water. This latter can be dealt with by suitable pH control measures. In Fig 1, these include a pH

sensor 13, and a reservoir 12 of a caustic. When the caustic is sodium hydroxide, that also serves to replenish sodium lost in the regenerative process.

[0030] It may be noted that chlorine (in the form of HOCl) in the regenerant water, having taken part in the reactions, then reverts back to chloride. Basically, the regenerant water, at the end of the ion-exchange and electrolysis cycle, is now in more or less the condition it was in before the ion-exchange took place. With provision for filtration /precipitation of impurities, and for adding small make-up quantities of its constituents, as necessary, the regenerant water is made ready to participate in further electrolysis reactions. In fact, the regenerant water can be recycled for an indefinite period. The amount of waste material generated by making the regenerant water suitable for recycling over long periods is tiny, whereby a comparison can hardly be made between disposing of this tiny amount of waste, and disposing of vast quantities of inadequately-treated ammonium-rich waste-water.

[0031] The regenerant water, being of high ionic strength, has a low electrical resistance. It is the regenerant water that becomes the electrolyte of the electrolytic cell 9, in the electrolysis station 24, whereby this low resistance assists in ensuring that the electrical energy requirements of the station are minimised.

[0032] The electrolytic cell preferably is structured with many anodes and cathodes, and the cell may be of divided configuration, in which membranes separate the anodic and cathodic compartments, or undivided. The cell electrodes can be configured in bipolar or mono-polar arrangement, as required. The anodes can be coated with a catalytic material (platinum or various metal oxides, for example) for promoting the desired reactions. The designer should aim to operate the electrochemical reactor at a current density such that the anode half-cell potential is maintained above 1.2 volts (with respect to a standard hydrogen electrode), to promote the anode reactions described above.

[0033] It may be noted that the regenerant water preferably should be driven down to very low concentrations of ammonium, in order for the regenerant water to be able to remove as much ammonium as possible from the ion-exchange resin in the column. The lower the concentration of ammonium in the regenerant water, the more effective the ion-exchange process can be, in flushing the ammonium ions out of the material in the columns 1,2,3, and into the regenerant water. Thus, it is preferred to circulate the brine (regenerant water) through the electrolytic cell and through the column continuously; then, as the electrolysis progresses, and the concentration of ammonium in the brine gradually diminishes, so the last remnants of ammonium stored in the sorbent can finally be captured. In some cases, it might be preferred to increase the pH of the brine to say ten; this shifts the redox equilibrium from soluble ammonia to ammonia gas, which can assist in extracting ammonium from the columns down to even lower levels.

[0034] Depending on the chemical characteristics of the waste-water stream, the regenerant water may pick up impurities, enough to affect the ammonium-flushing ability of the regenerant water, or to affect the efficiency of the electrolysis process, or otherwise to spoil the smooth operation of the treatment plant. The impure regenerant water may have to be periodically conditioned using precipitation and filtration of the regenerant to remove undesirable compounds. The amount of waste generated by this process would be small, particularly as compared with the problem of disposing of vast quantities of inadequately-treated ammonium-rich waste-water.

[0035] The ease may be noted with which the treatment plant can be automated. Fig 3 shows a basic system. Sensors may be used to monitor inlet and outlet ammonia concentrations, and it is recognised that it is adequate, in many cases, if the control of the whole system is based on these two measurements. More sophistication may be added, for example with sensors for monitoring the status of the electrolysis breakdown reactions, but still the sensors themselves are simple, and the actions resulting from the signals therefrom simply cause valves to open and close, to regulate flows and circulations through the system. The sensors may include e.g sensors 6,7 for ammonium concentration, pH levels, voltage and amperage magnitudes, and so on.

[0036] One of the important aspects of system control is to switch off the power to the cell when the reactions are complete. Of course, this should be done neither too early (which would leave some ammonium in the regenerant water), nor too late (which wastes electricity). The electrolysis time can be computed (having done pilot experiments to determine the operational characteristics of the cell), if the mass of ammonia/ammonium-N that has been sorbed into the regenerant water is known -- and, as mentioned, that mass can be computed, to a usually-adequate degree of accuracy, simply by comparing the ammonia concentration in the incoming waste-water stream with that in the out-going stream. The control system would compute the length of electrolysis time needed to remove that mass of ammonia. It will probably also be required to do pilot tests to determine the loading capacity of the ion-exchange columns, to complete the data needed to enable these computations to be done.

[0037] As mentioned, the incoming flow of waste-water need not be stopped when one of the columns has

become saturated, but is simply diverted to one of the other columns. Once the water has been electrolysed enough to deplete the ammonium content, the power to the cell is simply switched off. In some cases, sensors, as such, may not be needed, in that it is sufficient for the cell to be operated by a simple timer. Again, the length of time the cell should be energised can be determined by calculation, knowing the amount of ammonium transferred into the regenerant water.

[0038] Fig 2 shows how the apparatus of Fig 1 may be incorporated into e.g a municipal sewage treatment system. Apart from the ion-exchange station 23 and the electrolysis station 24, the other stations include grit removal 26, primary clarifier 27, sludge processing 28, activated sludge reactor 29, secondary clarifier 30, and UV treatment 32. It is preferred that the waste-water being treated be conducted through the ion-exchange columns after the waste-water has passed through the other stations, at least through those whereby suspended solids and dissolved organic material have been removed.

[0039] It should be noted that, since the ammonium is dealt with by the ion-exchange and electrochemical system as described herein, the other treatment stations in the municipal system can be designed for breaking down and removing organic contaminants from the water. In traditional treatment systems, some of the other oxidation stations sometimes have to be compromised, by having to provide habitation for aerobic microbes for ammonium treatment. Now, these stations, being relieved of the task of ammonium oxidation, can be optimised for the task of breaking down the organic contaminants.

[0040] It may be necessary to provide facility for further processing, such as chlorination, or UV treatment, for pathogen removal, which facility should preferably be placed after the ammonium treatment station.

[0041] The ion-exchange and electrochemical treatment system may alternatively be applied, in a municipal plant, as part of the sludge processing and dewatering circuit. The technology in this case would be used to remove ammonia from the supernatant water from those circuits, prior to return to the waste-water plant.

[0042] It should be noted also that traditional aerobic microbiological ammonium-oxidation stations take up a good deal of ground space. By contrast, the ion-exchange adsorption columns may be arranged vertically (i.e as packed-bed or fluidized-bed columns), thus permitting a saving in required space. The regenerant tank and the electrolytic cell do not take up much space, on the scale on which traditional aerobic stations are measured.

[0043] Other areas in which the ion-exchange-plus-electrochemical technology may be applied (other than main municipal water treatment facilities) include:-

- Treatment of effluent from sewage treatment lagoons or ponds. Sewage lagoons are often used by small communities for municipal waste-water treatment. The ion-exchange and electrochemical system could be used to remove ammonia from the effluent prior to discharge.
- Treatment of mine waste water contaminated as a result of blasting agents and other processing circuits.
- Treatment of effluent from food processing.
- Treatment of industrial waste-water, such as from factories making fertiliser products.

[0044] The systems as described herein may be summarised as follows. The ammonium is first sorbed out of the waste-water onto an ion-exchange medium, whereby the resulting discharged waste-water has an acceptably low ammonium content (typically, below one mg/litre of ammonia/ammonium nitrogen). When the sorbent is saturated, regeneration is done by ion-exchange, the ammonium being transferred from the sorbent into the regenerant-water. The regenerant-water is passed through an electrolytic cell, which breaks down the dissolved ammonium. In the cell, the regenerant-water is driven to the conditions in which nitrogen in the form of gas is thermodynamically favoured. The electrolysed, ammonium-diminished, regenerant-water can then be re-used in further regeneration episodes.

[0045] Some further points regarding the design of a treatment system that is to function in accordance with the invention will now be addressed.

[0046] The system as described herein can be used on a municipal scale, producing e.g 1800 kilograms of nitrogen per day; or it can be used to treat the waste-water stream from a house, or a few houses, prior to discharge into a lake or river, producing e.g one kg of nitrogen per day; or it can be used to treat the ammonium-rich waste-water stream from say a fertiliser factory, producing e.g 900 kg of nitrogen per day. In each case, in practising the invention, the electrolysis station 24 would be related to the ion-exchange station 23 on the basis that the electrolysis station is able to convert to nitrogen gas all the ammonium captured by the ion-exchange station.

[0047] In practising the invention, the ion-exchange and electrolysis stations should be matched to each other.

Where the ion-exchange or sorption station has just one column, it will be arranged that the one column is shut down, i.e. the waste-water stream is shut down, while the ion-exchange is being carried out. The waste-water stream is then restored, once all the ammonium has been captured out of the column, and transferred to the regenerant water. The regenerant water then passes, or continues to pass, through the electrolysis station, until all the ammonium in the regenerant water has been broken down. The designer must see to it that the electrolysis station can remove all that ammonium from the regenerant water before the column once again becomes saturated, i.e. before a fresh batch of ammonium enters the regenerant water.

[0048] As mentioned, it is preferred to keep the regenerant water circulating through the column at the same time that it is circulating through the electrolysis station. The benefit of this is that the regenerant water, as its concentration of dissolved ammonium is diminished, can then capture more of the residual ammonium from the ion-exchange media. One reason only one column is not preferred is that, with only one column, the one column has to be quickly put back on line, to minimise the interruption to the incoming waste-water stream; and of course it is not possible then to circulate the regenerant water through the column.

[0049] Usually, and preferably, it will be arranged that there is a plurality of columns (as in Fig 1), each one in an ion-exchange relationship with the body of regenerant water. The waste-water stream then can continue when one column becomes saturated, the stream simply being routed through one of the other columns. Where the electrolysis station has to cope with the ammonium from several columns, the designer of course should see to it that the electrolysis station can break down the ammonium in the body of regenerant water faster than the ammonium is being fed into the body of regenerant water in aggregate from the several columns.

[0050] In a case where the waste-water stream is not to be shut off (in a municipal treatment system, for example), preferably three ion-exchange columns (or beds) are provided. Just one body of regenerant water is provided, and the system can be set to direct the body of regenerant water through each of the columns sequentially. Just one electrolysis station is provided. During electrolysis, the body of regenerant water preferably is circulated continuously through the one column and through the electrolysis station.

[0051] The ion-exchange columns (or beds) have to be sized for the anticipated through-flow of ammonium water, both in terms of volumetric flow of water, and in terms of the ammonium content of the waste-water. In a typical case, the through-flow of water may be 1000 cubic metres per day, having an ammonia/ammonium-N content of twenty grams per litre (i.e. an ammonia/ammonium-N stream of fifteen kg per day). For such a case, the designer might provide three columns, each column containing e.g. 4000 kg of zeolite.

[0052] As described, the invention is aimed at capturing ammonium from a waste-water stream into a body of brine or other regenerant water, by ion-exchange; then, as described, the brine passes through an electrolytic cell, where the ammonium is broken down directly to nitrogen gas. It is recognised, as described, that such a system can be designed so that the brine regenerant water is hardly changed, chemically, by this overall cycle, whereby the body of regenerant water can remain in use for long periods without a need for the ion-exchange salts to be continually replenished. Thus, apart from minor inputs of chemicals for pH-control purposes, the only major input required into the system is the electrical energy (supplied through rectifier 11) used in the electrolytic cells 9.

[0053] In a treatment system handling say fifteen kg of ammonia/ammonium-N per day, the designer should see to it that the electrolytic cell(s) are capable of converting that whole stream to nitrogen gas. An electrolysis station capable of handling that quantity of nitrogen would typically be operated at a current density of between 300 and 1000 amps per square metre of the anode.

[0054] As mentioned, the electrolysis station must convert the nitrogen to gas at a faster rate than the rate at which ammonium-N is being captured out of the ion-exchange columns. Thus, the electrolysis station, in a typical case, might have to oxidise the fifteen kg of nitrogen in less than one day. In that case, a current in the region of 3500 amps would be needed, with a desired operating voltage of less than four volts.

[0055] The capacity (i.e. volume of electrolyte) of a suitable electrolytic station typically would be around xxx litres. Typically, where the column has a water capacity of xxx litres, and the electrolytic station can contain xxx litres of electrolyte, the volume of the body of regenerant water would be xxx litres. The tank for containing the regenerant water preferably should be large enough to contain the whole body of regenerant water.

## Claims

- Claim 1.** Apparatus for diminishing the concentration of ammonium in waste-water, including:  
[02] an ion-exchange medium, and conduits for conducting the flow of waste-water therethrough, whereby ammonium in the waste-water is sorbed thereon;  
[03] an operable means for ceasing the flow of waste-water through the medium, when the ion-exchange medium is loaded with ammonium, and for passing a volume of regenerant-water through the medium, whereby the ammonium in the medium is transferred to the regenerant-water;  
[04] an electrolysis station, and conduits for passing the volume of regenerant-water therethrough;  
[05] wherein the electrolysis station is so configured as to oxidise the ammonium in the regenerant water, and as to thermodynamically favour the transformation of the nitrogen component thereof to nitrogen gas.
- Claim 2.** Apparatus for diminishing the concentration of ammonium in waste-water, and for disposing of the ammonium as nitrogen gas, wherein:  
[02] the apparatus includes an inlet port, for receiving a flow of waste-water to be treated;  
[03] the apparatus includes a discharge port, for discharging treated waste-water;  
[04] the apparatus includes a sorbent-container, in which is contained an sorbent-body, which is effective to sorb ammonium from water passing therethrough;  
[05] the apparatus includes a regenerant-tank, containing regenerant-water;  
[06] the regenerant-water contains, in solution, a regenerant-substance, which is of such nature, and of such concentration, as to have an ion-exchange relationship with respect to ammonium ions sorbed onto the sorbent-body;  
[07] the apparatus includes a system of connecting conduits and operable flow valves;  
[08] the valves are operable in a sorption-mode, and in a regenerant-mode;  
[09] in the sorption-mode, the valves are arranged so that waste-water passes from the inlet port, through the sorbent-container, and out of the discharge port;  
[10] in the regenerant-mode, the valves are arranged so that regenerant-water from the regenerant-tank passes through the sorbent-container, and back to the regenerant-tank;  
[11] the apparatus includes an electrolytic cell, which is structured for containing water undergoing electrolysis, and includes an anode and a cathode;  
[12] the cell is so arranged that, upon ammonium-rich water being present in the cell, the ammonium is broken down, in such manner that the nitrogen component thereof takes the form of nitrogen gas;  
[13] the cell includes a gas-vent, through which nitrogen gas created in the cell can be vented from the cell;  
[14] the apparatus is so arranged that the regenerant-water passes through the cell, and undergoes electrolysis, whereby ammonium in the regenerant-water is broken down to nitrogen gas, and the concentration of ammonium is thereby diminished.
- Claim 3.** Apparatus of claim 2, wherein:  
[02] in the sorption-mode, the valves prevent the regenerant-water from entering and passing through the sorbent-container;  
[03] in the regenerant-mode, the valves prevent the waste-water from entering and passing through the sorbent-container;
- Claim 4.** Apparatus of claim 2, wherein the regenerant-substance has an ion-exchange relationship with respect to ammonium ions sorbed onto the sorbent-body, in that, upon the regenerant-water passing through the sorbent-body, the regenerant-substance is sorbed onto the sorbent-body, thereby displacing ammonium ions from the sorbent-body, the displaced ammonium ions being taken into solution in the regenerant-water.
- Claim 5.** Apparatus of claim 2, wherein:  
[02] the regenerant-tank is physically separate from the electrolytic cell;  
[03] conduits connect the regenerant-tank with the cell;  
[04] an operable pump is effective, when operated, to circulate regenerant-water through the cell.
- Claim 6.** Apparatus of claim 2, wherein the sorbent body is so structured and arranged as to sorb all but a negligible concentration of ammonium from the waste-water.
- Claim 7.** Apparatus of claim 2, wherein the waste-water being treated passes through the apparatus without



itself being subjected to electrolysis.

- Claim 8. Apparatus of claim 2, wherein:  
[02] the adsorption-container comprises a plurality of separate containers, containing respective bodies of sorbent material;  
[03] the apparatus includes operable flow-control means, so arranged that the flow of waste-water can be directed first through one of the containers, until that container becomes loaded with ammonium, and then the flow of waste-water can be diverted and directed to a next one of the containers.
- Claim 9. Apparatus of claim 8, wherein the configuration of the apparatus is such that:  
[02] during regeneration of one of the containers, the body of regenerant water can be passed through that container;  
[03] the body of regenerant water can continue to circulate through that container, while electrolysis of the body of regenerant water continues.
- Claim 10. Procedure for diminishing the concentration of ammonium in a flow of waste-water, including:  
[02] passing the flow of waste-water through an ion-exchange medium, whereby ammonium in the waste-water is sorbed thereon;  
[03] when the ion-exchange medium is loaded with ammonium, ceasing the flow of waste-water through the medium, and passing a volume of regenerant-water through the medium, whereby the ammonium in the medium is transferred to the regenerant-water;  
[04] passing the volume of regenerant-water through an electrolysis station;  
[05] wherein the electrolysis station is so configured as to oxidise the ammonium in the regenerant water, and as to thermodynamically favour the transformation of the nitrogen component thereof to nitrogen gas.
- Claim 11. Procedure for diminishing the concentration of ammonium in waste-water, and for disposing of the ammonium as nitrogen gas, including:  
[02] providing an apparatus that falls within the scope of claim 1;  
[03] conducting the flow of ammonium-laden waste-water in through the inlet port;  
[04] passing the waste-water through the sorbent body, thereby removing ammonium from the waste-water;  
[05] discharging the treated waste-water through the discharge port;  
[06] when the sorbent body is laden with sorbed ammonium:-  
[07] - shutting off the sorbent body from the flow of waste-water;  
[08] - passing the regenerant-water from the regenerant-tank through the sorbent-container, and back to the regenerant-tank, thereby breaking down the ammonium in the regenerant-water;  
[09] - then re-opening the sorbent body to the flow of waste-water;  
[10] removing the ammonium from the regenerant-water, by:-  
[11] - passing the regenerant-water through the electrolytic cell;  
[12] - applying electrical energy to the cell, whereby the ammonium in the regenerant-water breaks down;  
[13] - returning the regenerant-water to the regenerant-tank.
- Claim 12. Procedure of claim 11, including:  
[02] monitoring the ammonium content of the incoming waste-water, and of the outgoing waste-water, over a period of time, as the waste-water passes through the sorbent-container;  
[03] from the thus-derived information, computing the quantity A kilograms of ammonium in the absorbent material;  
[04] computing a resultant quantity AR kilograms of ammonium that is present in the regenerant water, after ion-exchange;  
[05] pre-preparing look-up tables, from the electrolytic characteristics of the cell, of time-durations appropriate to breaking down different quantities of ammonia in the regenerant water;  
[06] looking up the appropriate time period P for which the cell should be operated, to break down the quantity AR of ammonium;  
[07] operating the cell for that time period P.
- Claim 13. Procedure of claim 11, including sensing when the sorbent body is laden with sorbed ammonium by monitoring the concentration of ammonium in the waste-water passing therethrough, and automatically initiating regeneration episodes in response.
- Claim 14. Procedure of claim 11, including monitoring the concentration of ammonia in the outgoing-waste-water, and operating the ion-exchange station, to regenerate same, responsive to that



concentration going above a pre-set threshold.

Abstract of the Disclosure

Title: ION-EXCHANGE / ELECTROCHEMICAL TREATMENT OF AMMONIA IN WASTE-WATER

The ammonium is first sorbed onto an ion-exchange medium. When the sorbent is saturated, regeneration is done by ion-exchange, the ammonium being transferred into the regenerant-water. The regenerant-water is passed through an electrolytic cell, which breaks down the dissolved ammonium. The cell is operated to drive the regenerant-water into conditions where nitrogen in the form of gas is thermodynamically favoured. The electrolysed, ammonium-diminished, regenerant-water can be re-used in further regenerant episodes.

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Docket: 616-75

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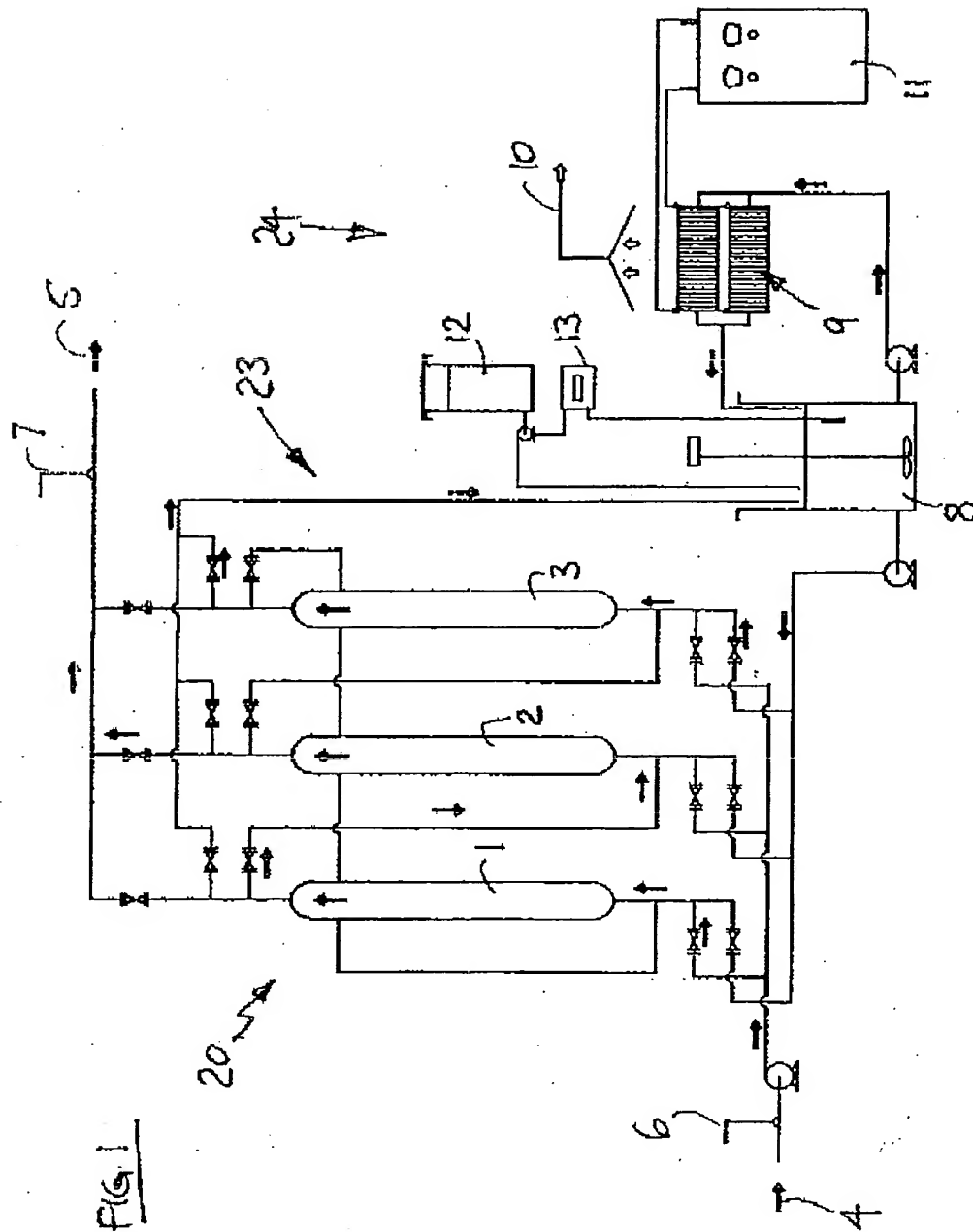
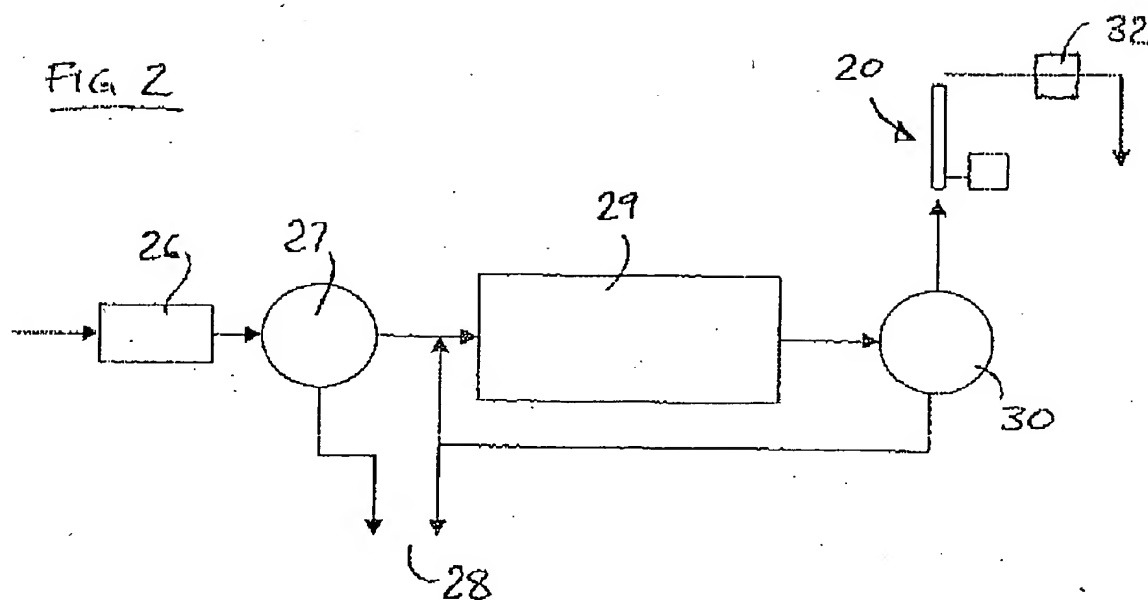
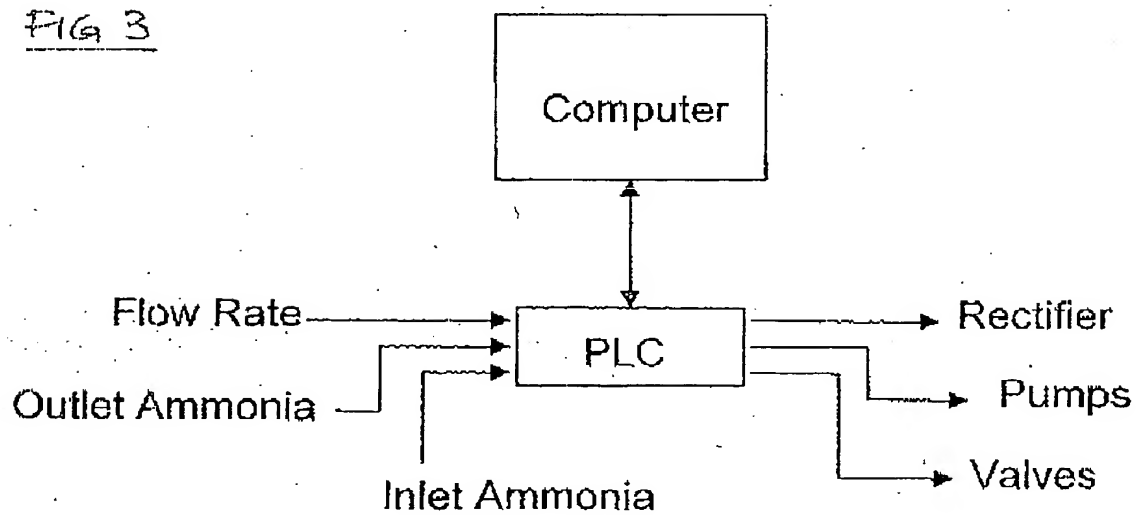


FIG 1



FIG 2FIG 3

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